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CAT.33 Code 217

NASA W56592

Contract NAS 8-11066
Control No. NASA 110-10320

STUDY OF ADHESION AND COHESION
IN VACUUM

Third Quarterly Report

1 April 1964

Materials Technology Department
Components and Materials Laboratory

AEROSPACE GROUP

HUGHES

HUGHES AIRCRAFT COMPANY
CULVER CITY, CALIFORNIA

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HUGHES AIRCRAFT COMPANY
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MATERIALS TECHNOLOGY DEPARTMENT
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STUDY OF ADHESION AND
COHESION IN VACUUM

by

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Contract Number NAS 8-11066

George C. Marshall Space Flight Center
Huntsville, Alabama

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FOREWORD

This report was prepared by the Hughes Aircraft Company to cover work completed during the period 1 January to 31 March 1964, under a NASA contract to study adhesion and cohesion of metals in vacuum. This contract is sponsored by the George C. Marshall Space Flight Center, NASA, Huntsville, Alabama with Mr. Keith Demorest as Project Engineer.

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ABSTRACT

During this report period, the principal effort was directed toward the development of a suitable method for the measurement of the contact and separation loads of the adhesion and cohesion test specimens. Problems associated with the strain link on which semiconductor strain gages were mounted were not resolved completely and therefore, optimum load measuring techniques were not perfected. A re-designed strain link which will be isolated from transient thermal gradients is expected to solve this problem. Preliminary tests of the new strain link are very encouraging. In a number of cohesion experiments with copper test specimens at 5×10^{-9} torr and 500°C it was found that there was no cohesion up to 1000 seconds at a load of 80% at the compressive yield strength, but cohesion was detected at 125% of the compressive yield strength in 10000 seconds.

Except for the load measuring difficulties, the test apparatus has performed excellently and has met the design goals of 5×10^{-9} torr with the test specimens operating at a temperature of 500°C .

INTRODUCTION

The purpose of this program is to determine the temperature and time conditions under which adhesion or cohesion of structural metals in a vacuum occurs. The data developed from this study will provide spacecraft designers with engineering data required to insure separation of instrument capsules and other components from spacecraft which have been exposed to a hard vacuum.

The program includes design and fabrication of a vacuum test chamber that will (1) provide an environmental pressure not greater than 5×10^{-9} torr, (2) contain a loading device capable of providing and measuring tensile and compressive pressures of from 0 to 100,000 psi and (3) provide a range of testing temperatures from 25°C to 500°C.

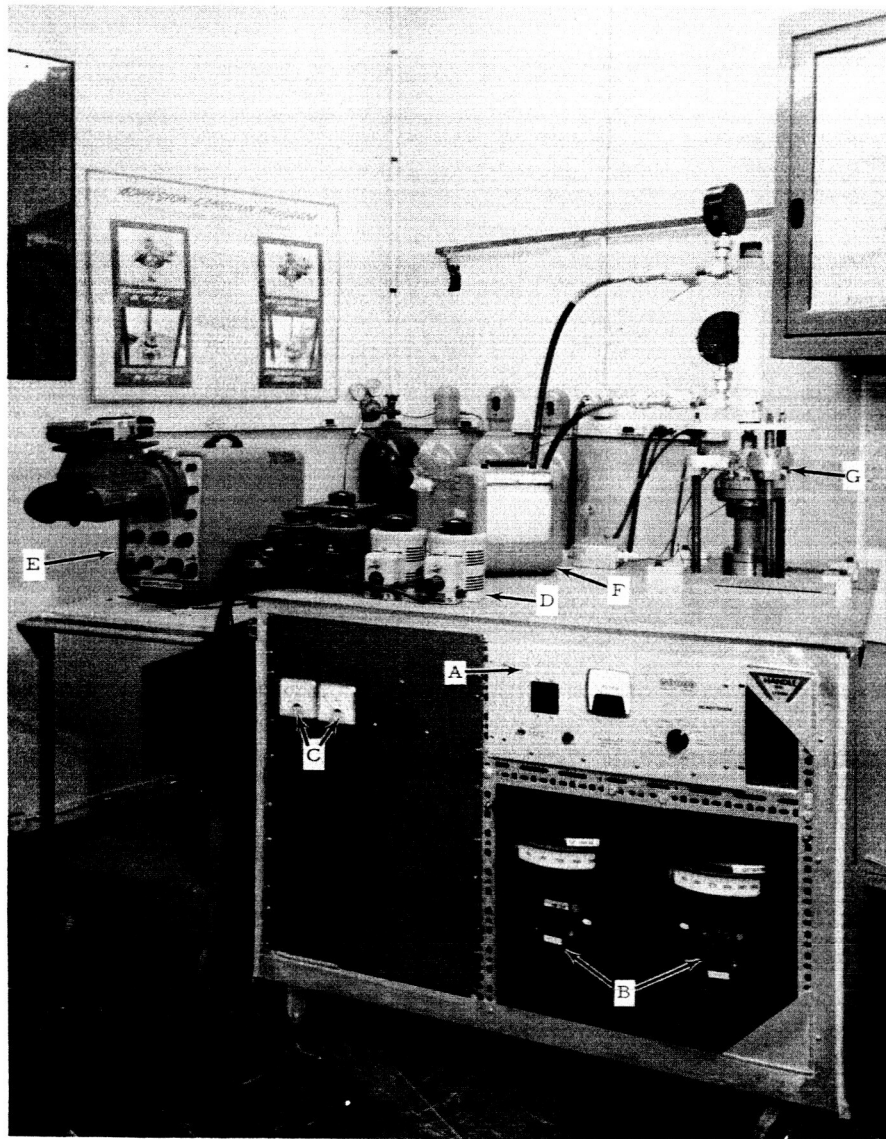
Tests will be made by application of compressive loads to contacting test specimens in the vacuum chamber for a given time period. Then the tensile force required to separate the two test specimens will be measured to determine the extent of adhesion or cohesion.

Thirteen different combinations of metal couples will be evaluated. Material screening tests will be performed first under the most severe environmental conditions of contact pressure and temperature to allow concentration of effort upon those couples that will cause the most difficulty. Statistical analysis of the data from factorial experiments with the materials will yield behavior equations with known confidence limits that designers may use in an engineering sense to determine whether adhesion of contacting surfaces will occur. If the conditions causing adhesion cannot be altered to avoid adhesion, the behavior equations can be used to predict the amount of force required to separate the joint. It is anticipated that the statistical approach to the design of the experiments and analysis of the results will reduce the number of tests to a minimum.

TEST APPARATUS

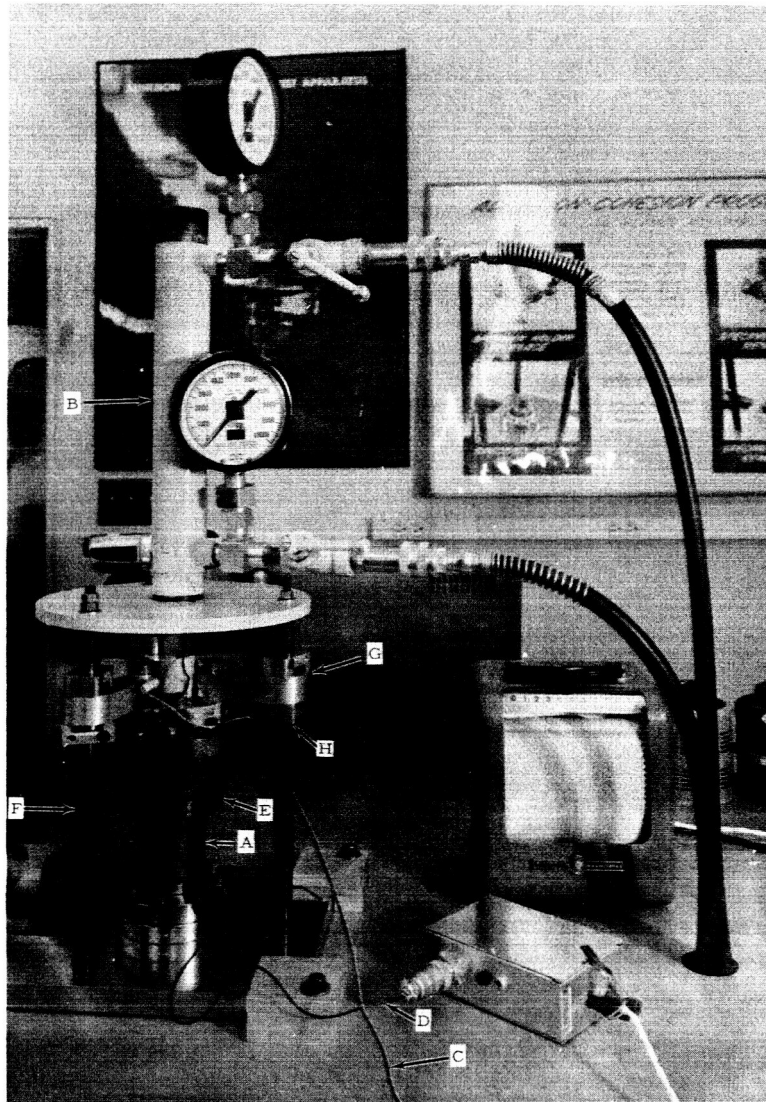
The design of the test apparatus was shown and its operation was described in the First Quarterly Report. Photographs of the apparatus with captions listing the various components are shown in Figures 1 through 6. Figure 1 shows the completely assembled apparatus. The vacuum chamber and loading mechanism are shown in Figure 2.

Figures 3 and 4 show the strain link flange assembly disassembled from the apparatus. In Figure 3 the specimen holder is in place and in Figure 4 the specimen holder has been removed. Figures 5 and 6 are similar views at the lower specimen mount disassembled from the vacuum chamber. In Figure 5, the specimen holder is in place as used in testing. The specimen holder has been removed in Figure 6 to permit viewing of the individual components.



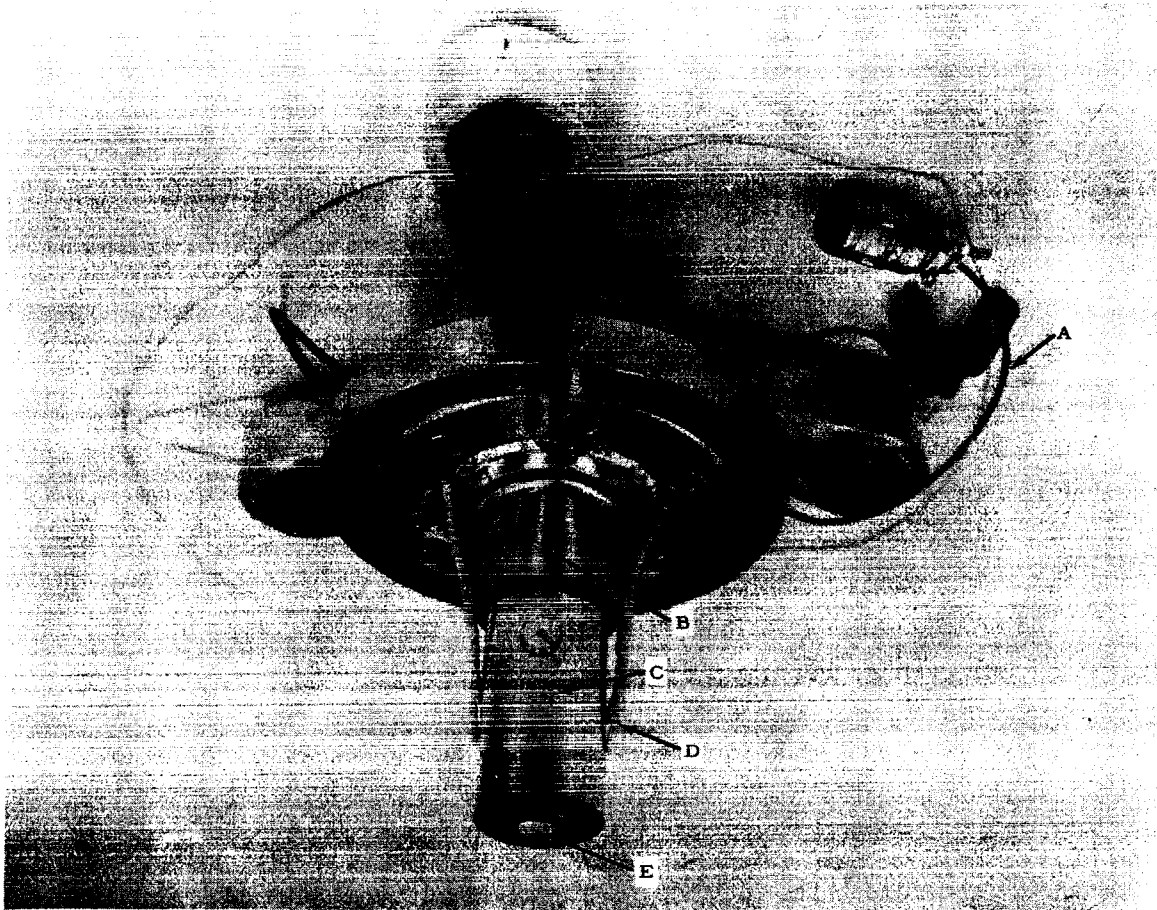
- A. Control panel for Ultek 100 liter/sec Vac-Ion pump
- B. Controlling pyrometers for test specimens
- C. Voltmeter and ammeter for specimen heater
- D. Variacs for regulating power to specimen heaters and bake-out heaters
- E. Oscilloscope for measuring strain gage voltage
- F. Varian G11A recorder for recording strain gage voltage
- G. Vacuum chamber and loading mechanism

Figure 1. Adhesion and cohesion test apparatus.



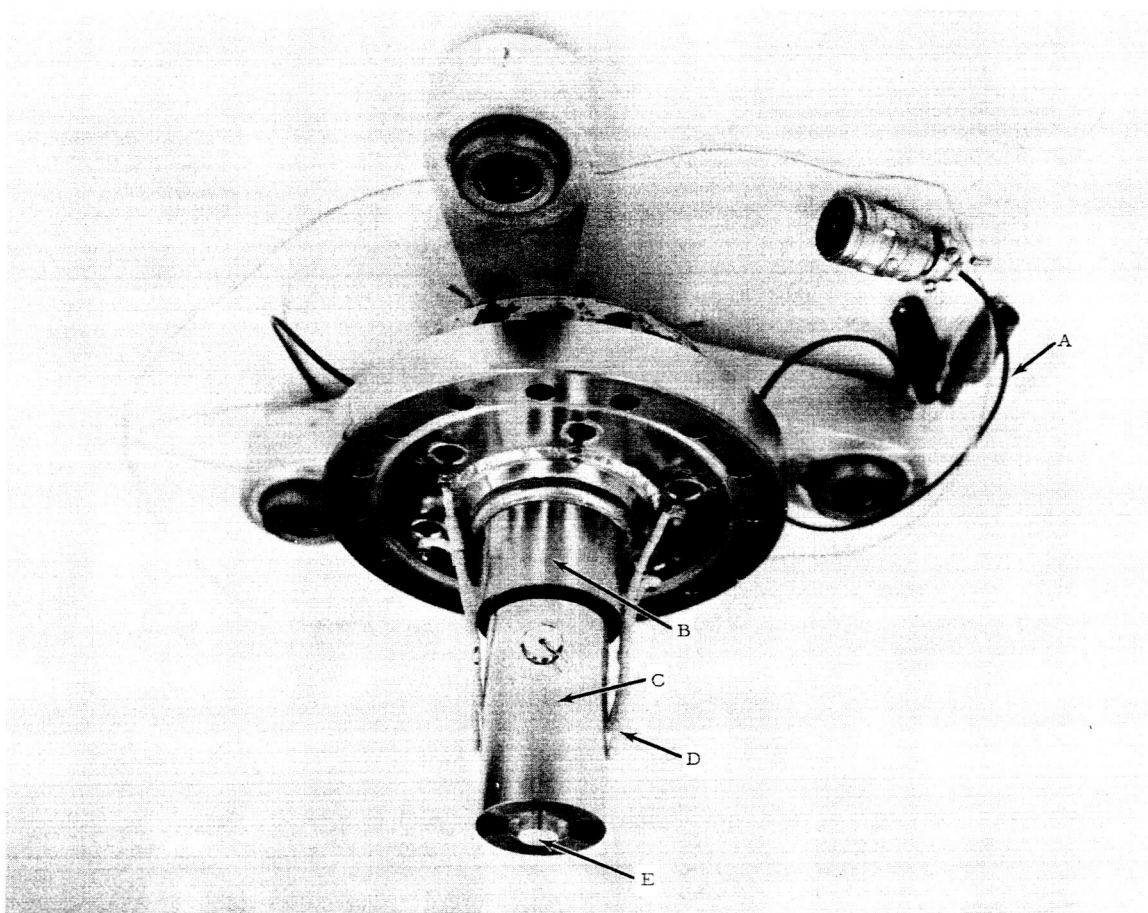
- A. Vacuum chamber
- B. Double acting hydraulic ram
- C. Thermocouple leads for upper specimen
- D. Strain gage leads
- E. Bellows
- F. Guide rod
- G. Bearing support arm
- H. Stop for holding specimens separated

Figure 2. Vacuum chamber and loading mechanism.



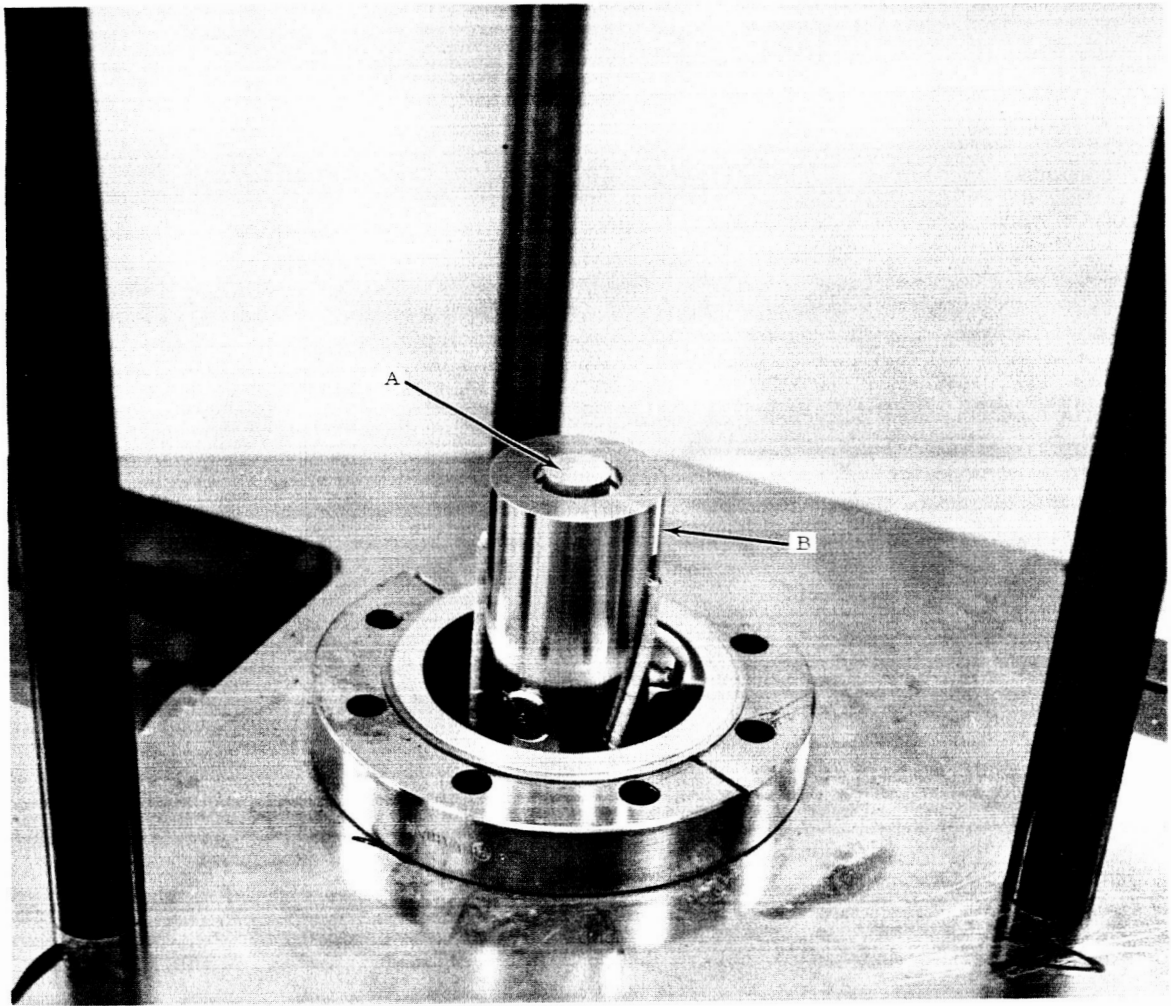
- | | |
|---------------------|------------------|
| A. Strainlink leads | D. Thermo couple |
| B. Strain link | E. Test specimen |
| C. Specimen holder | |

Figure 3. Strain link flange assembly.



- | | |
|---------------------|------------------|
| A. Strainlink leads | D. Thermo couple |
| B. Strain link | E. Test specimen |
| C. Specimen holder | |

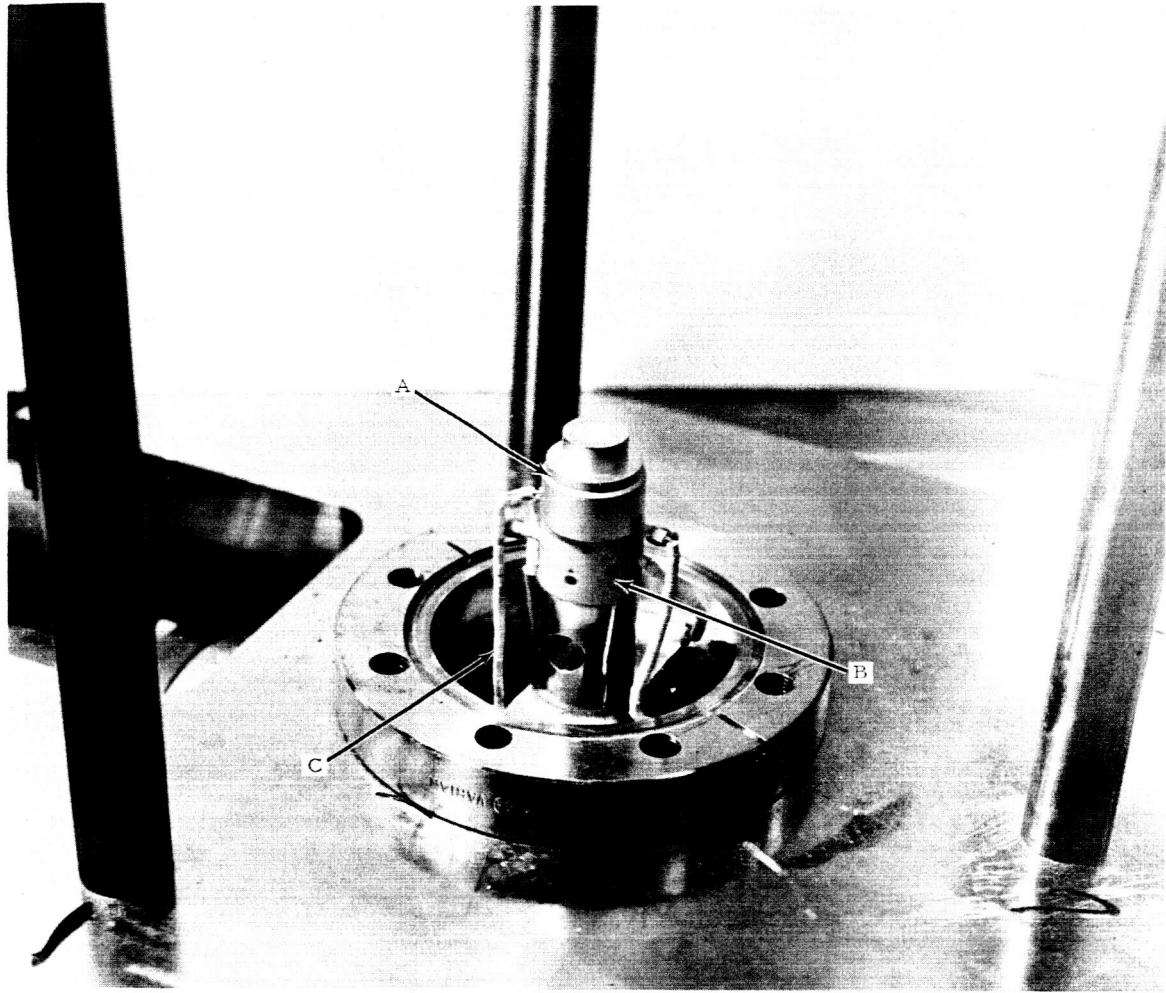
Figure 3. Strain link flange assembly.



A. Test specimen

B. Specimen holder

Figure 5. Lower specimen mount.



A. Test specimen
B. Specimen heater

C. Thermo couple

Figure 6. Lower specimen mount with specimen holder removed.

EXPERIMENTAL PROGRAM

During this period, experiments were performed to determine the cohesion behavior of copper to copper couples at a temperature of 500°C. The test specimens were of the design shown in Figure 2 of the First Quarterly Test Report. The contacting surfaces of the test specimens were ground to a 27 to 37 RMS finish and were vapor degreased per MIL-S-5002. The test apparatus was as described in previous quarterly reports.

The system was tested for its vacuum capabilities with the specimens operating at the maximum temperature of 500°C. Temperatures were controlled by Minneapolis-Honeywell Brown Pyro Vane pyrometers connected to individual chromel-alumel thermocouples inserted in each specimen. The environmental pressure was measured by the ion pump gage. Under these conditions with the test specimen separated, the pressure goal of 5×10^{-9} torr was achieved. Once the tantalum resistance elements of the heaters were initially outgassed, they added very minor gas load to the system. The heaters performed very satisfactorily with the exception of a heater lead burning out by accidental shorting against the specimen holder in one test. By decreasing the length of the ceramic insulators on the leads to give better flexibility of the leads, the possibility of cracking the insulators and causing shorts was minimized. After this change, no further heater failures occurred.

Kulite-Bytrex MP-102-6 semiconductor strain gages were mounted on the strain link in accordance with the system described in the Second Quarterly Report. The Budd Instrument Model HW-1 strain indicator used to record the test loads was found to have insufficient stability to utilize the sensitivity of the semiconductor gages. To overcome this problem, a full resistance bridge was constructed to accomodate the strain gages and the strain signals were fed into a dynamic recording system. The recorder used is a Model G 11a Varian Associates recorder.

The utilization of semiconductor strain gages to measure loads has been the most formidable problem during this quarter and consequently quantitative measurements of breaking loads of cohered specimens were not obtained. The difficulties encountered with strain gages and the corrective action for overcoming these problems is elaborated upon in the section "Load Measuring Instrumentation."

The double acting hydraulic ram for application of loads and for separating the specimens operated satisfactorily in short term tests, but lost pressure in the long term tests. This was due to the switching valve not providing positive closing action which permitted the hydraulic fluid to leak back into the reservoir. This condition was corrected by the installation of additional valves in the hydraulic lines so that when the desired pressure in the hydraulic ram was reached the valves could be closed and the pressure could be held constant. To prevent pressure surges and consequent jerky movement of the ram in the hydraulic system during pumping, the "high pressure" chevron seal was replaced by an "O" ring seal. This provided much smoother movement of the piston during pumping.

In operation of the system, the test specimens are installed, and the vacuum system is closed. Two liquid nitrogen-cooled sorption pumps are connected by glass tubing to the test unit. The sorption pumps are operated sequentially to lower the pressure to the 10^{-4} torr range. The 100 liter/sec. ion pump is then started and a glass seal-off from the sorption pumps is made. When the pressure reaches 10^{-5} torr, system bakeout is commenced by the use of glass cloth heating tapes which heat the system to at least 350°C . The specimen heaters are also operated at test temperature during bakeout. One to two hours is usually required for bakeout. When the pressure starts dropping, the heating tapes are turned off and the system is operated until the pressure reaches 5×10^{-9} torr or less. After the specimens have remained separated overnight (a minimum of 6 hours) at the minimum environmental pressure, the adhesion and cohesion tests are commenced.

The sequence of testing the copper specimens was to bring the specimens in contact and apply the compressive load. The load was applied successively for time periods of 10, 100, 1000, 10000, and 60000 seconds or until measurable cohesion occurred. When cohesion occurred, the test for this set of specimens was concluded. After each test in the sequence, the specimens were held in the separated position for 30 minutes prior to applying the load for the next time period.

As mentioned previously, satisfactory load measuring techniques have not yet been developed. The data on the tests that have been run during this quarter are shown in Table I.

TABLE I.

Copper Cohesion Experiments Run at
500°C at a Pressure of 5×10^{-9} torr

Specimen Number		Results
Top	Bottom	
1	2	Bottom heater lead burned out prior to test.
3	4	Due to malfunction of strain gage system, specimens were severely overloaded and cohesion occurred in 100 seconds
5	6	No welding observed in exposures of 10, 100, and 1000 seconds. Hydraulic pressure did not hold for the 10000 second exposure. Upon disassembly of system the appearance of the test specimens indicated that their compressive yield strength had been exceeded slightly, which indicated that the strain gages were reading too low.
7	8	Due to unsatisfactory response of the strain gages, the load was applied by substituting a known overload of atmospheric pressure acting on the vacuum system in place of the hydraulic pressure. This load amounted to 125% of the estimated compressive yield strength of the copper. In periods of 10, 100, 1000, and 10000 seconds no measurable cohesion occurred as evidenced by no increase in hydraulic pressure in the hydraulic separating device. In the 60000 second exposure, definite cohesion occurred as determined by increased hydraulic pressure to separate the specimens and visual appearance of specimens which were indented by the specimen holders when tensile force was applied to the welded or adhered specimens.
9	10	Due to malfunction of strain gage system, the specimens were severely loaded beyond their yield strength. Cohesion occurred in a 10 second exposure.
11	12	Tests were run in the same manner as test specimens 7 and 8. No cohesion was detected in periods of 10, 100, and 1000 seconds. Some cohesion was detected in 10000 seconds, but a quantitative measurement was not obtained.

LOAD MEASUREMENT INSTRUMENTATION

The measurement of both the tensile forces required to separate bonded specimen couples and the compressive forces needed to apply the loads is accomplished with a semiconductor strain link. The strain link is calibrated in compression and tension with an Instron Universal Testing Machine. In the original design, this load cell was incorporated in the top vacuum flange. This design sensed forces on the specimen couples independent of drag on the apparatus. Due to difficulties encountered in the instrumentation of this strain link, this design was modified and an independent strain link is being fabricated which will be inserted between the hydraulic ram and the vacuum flange.

A. SEMICONDUCTOR STRAIN GAGE PROBLEMS

The original difficulty encountered with the Kulite-Bytrex semiconductor strain gages was the inability to obtain a satisfactory bond to the strain link. The strain gage supplier indicated that the mounting and techniques would be similar to those used for wire or foil gages and therefore, no problems were anticipated and no extra precautions were taken. However, a number of sets of gages were attempted by experienced Hughes personnel without success. While it initially appeared that the gages were properly mounted and the resistance remained constant, the strain from small loads could not be detected and when large loads were applied, the gages would not return to zero strain upon removal of the load. Thus, it appeared that the mounting techniques were not suitable.

After considerable time was consumed in attempting to solve this problem, it was decided to engage Micro-Systems, Inc. (Pasadena, California) to design and install a load cell using the existing strain link. Their design also utilized semiconductor strain gages. Micro-Systems, Inc. are specialists in this field and have had a successful record of designing load measuring systems for Hughes. Their first attempt resulted in a failure similar to those Hughes personnel had encountered.

During the second instrumentation cycle, Micro-Systems had an accident with the strain link. The link was loaded beyond the design load which caused permanent set and deformation in the link. It was necessary to re-fabricate this assembly (at Micro-Systems expense) by machining out the deformed portion and rewelding the new link into the vacuum flange. Through close cooperation with the machining vendor this job was expedited and the re-fabrication was completed in two days.

On their third attempt on the newly fabricated link, the mounting of the gages was successful. The gages sensed the loads perfectly at room temperature, but upon applying heat to the test specimens, the gages behaved in an unstable manner. Large fluctuations in the zero balance point occurred at elevated temperatures. After several days of trouble shooting, Micro-Systems personnel attributed the erratic performance to thermal stresses introduced in the strain link from thermal gradients created by the heat cycling. However, when the entire apparatus was covered with insulation and heat soaked for three days to attain thermal stability the result was still no improvement in the strain gage performance. Since the drift in the strain link response represented a signal larger than the 80 lb. load which was required to be measured, it was necessary to make modifications to correct this problem.

It should be emphasized that the temperature capability of the strain gages was not exceeded. The gages are designed to withstand 200°C for long term exposure and this temperature was not exceeded. The instability was more likely due to mechanical instability of the strain link from erratic thermal expansion. It was decided the best solution to this problem was to isolate the strain link section from the heat zone. A new strain link has been designed. This link will be inserted between the top loading flange and the hydraulic ram. Figure 7 is a drawing giving the dimensions of the new strain link.

The primary difference between the new method of measurement and the original is that the zero reference or check point is moved. With the original internal strain link, the only strain changes measured

were those due to loads on the specimen. However, with the new design, the strain link connects the hydraulic ram with the top flange and the link is under a tensile load due to the atmospheric pressure acting on the top flange when the system is under vacuum and the specimens are separated.

It is expected that it will be necessary to use the vacuum load as a zero reference. Thus when the compressive force is applied to the specimens, the strain link senses the differential between the initial tensile load acting on the strain link and the compressive force acting on the specimens.

B. SEMICONDUCTOR STRAIN GAGES

The semiconductor strain gage operates on the same basic principal as the wire or the foil gage, except that germanium or silicon crystal wafers are employed because of their Piezo-resistive effects. The changes in resistance of these gages upon load application are from 65 to 100 times as great as those produced with wire and foil gages. This type of gage enables accurate measurements at extremely low as well as high loads. The gages are designed to withstand 200°C for long term exposure and 285°C for very short periods. Gages are guaranteed over a range of ± 3000 microinches/inch of strain, however many of these units have been used to the 5-6,000 microinches/inch range.

C. CIRCUITRY

The circuits used for these measurements are very simple and basically consist of a wheatstone bridge made up of four fixed resistances (the four semiconductor gages) and an adjustable resistance (potentiometer) for balancing the bridge. Two of the gages are set on the load sensor parallel to the direction of strain for strain measurement and two gages are set perpendicular to the load sensor. The latter gages are used as dummies or temperature compensators. The wheatstone bridge is driven by a constant current transistorized power supply which in turn is driven by 5-6 volt dc batteries. The output of the bridge is recorded on a

Varian G-11A recorder which has a 10 millivolt full scale deflection on 5 inch paper. The recorder may be attenuated to read voltages up to 100 volts scale. A schematic diagram of the circuit is shown in Figure 8.

D. OPERATIONAL PROCEDURE

The bridge is balanced to a null point indicating zero load on the strain link. The hydraulic ram is positioned so that the strain link measures the force required to keep the specimens separated. Compressive loads applied are measured by the strain link when the specimens are in contact. Tensile loads required to separate specimens which show signs of adhesion or cohesion are considered to be those forces in excess of that required to originally keep the specimens apart. Mathematically, the various loads may be defined as follows:

Load on strain link when specimens are separated = $k(\epsilon_t - \epsilon_n/)$

Compressive Load on specimens = $k(/ \epsilon_t - \epsilon_n/ + / \epsilon_n - \epsilon_c/)$

Tensile load to separate specimens = $k(/ \epsilon_t - \epsilon_n/ + / \epsilon_1 - \epsilon_t/)$

where ϵ_t = Strain on the link due to pressure differential of the vacuum

ϵ_c = Strain on the link due to compressive force applied to the couple

ϵ_1 = Strain on the link due to tensile forces applied to the couple

ϵ_n = Strain reading of the link at the null balance point

k = Constant

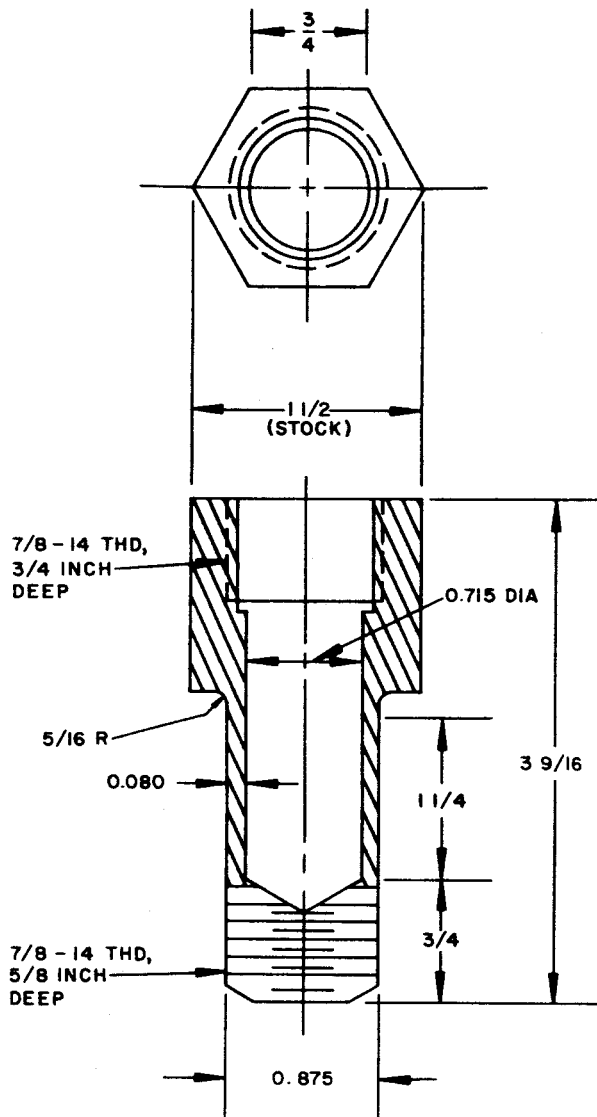


Figure 7. Dimensions of strain link.

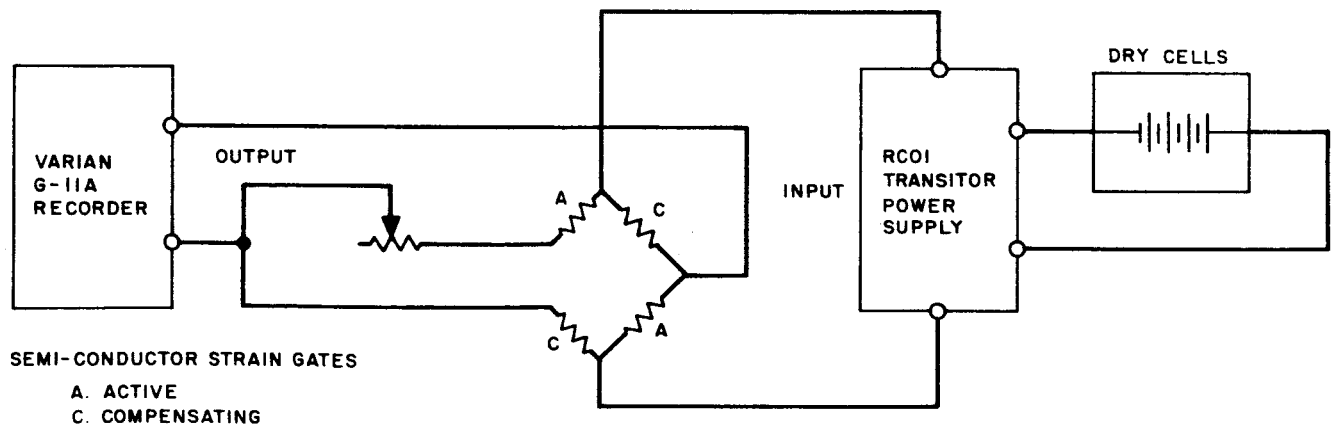


Figure 8. Schematic diagram of load sensor apparatus.

WORK PLANNED FOR NEXT QUARTER

A re-designed strain link which will isolate the strain link from transient thermal gradients caused by cycling of the test specimen heaters will be manufactured and installed. Semiconductor strain gages will be mounted on the strain link and the gages will be calibrated. Overtime effort will be expended to expedite the program. From a thorough analysis of the previous strain gage problems, a high degree of confidence is held that the re-designed strain link will provide a solution to the measurement of loads. The adhesion and cohesion experiments will then be continued.

PROPOSED FOLLOW-ON PROGRAM

At the request of Mr. Keith Demorest, Project Engineer, a proposed follow-on program for the study of adhesion and cohesion in vacuum was submitted on 4 February 1964 to George C. Marshall Space Flight Control, NASA, Huntsville, Alabama.

It is anticipated that adhesion or cohesion may not occur with some metal to metal couples under simple static loading and the environmental parameters specified in the current program.

Since loads between mating components of spacecraft are generally more complex than simple compression loads, it is logical that the effects of other types of loading upon adhesion and cohesion be studied. For example, vibrations caused by rocket engine firing or sliding motion of one component upon another where mating surfaces are not parallel to each other would cause abrasion of contacting surfaces. Such abrading action would tend to break up natural oxide or barrier films thereby promoting bare metal contact. Thus, it would be expected that under vibrating loads, the onset of adhesion or cohesion would occur at levels of time, temperature, and contact pressure lower than those that cause adhesion or cohesion with static loading. From these considerations, a follow-on program with the additional parameter of vibratory loading was proposed. This expansion of the current program will utilize the special test equipment which was designed and constructed under the current contract, but will require equipment modification to permit the application of vibratory loads. Other test parameters and the test materials will be essentially the same as those being used in the current program. The philosophy at the testing sequence was to make initial tests at the parameters below those at which bonding took place under static loads. By progressive variation of the test parameters, the threshold at which adhesion or cohesion of the various metal couples under vibration loading occurs will be established. The data will be presented in appropriate form to permit spacecraft designers to establish design requirements for separation of components after they have been subjected to high temperatures, low environmental pressures, and mechanical vibration.

ACKNOWLEDGEMENTS

D. V. McIntyre and W. P. Weber assisted in the experimental program.